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Tentative Procedure

Method 4-A S

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Determination of "T" in Sludges from the Disposal Plant
Settling Tank

REPORT NO.

KZ 1351

Sample Size and Preparation:

Weigh 25 grams of the well-mixed sample into a 600 ml. beaker, dilute to 300 ml. with distilled water, stir well and neutralize with HCl. Allow sample to leach over night or at a low heat with frequent stirring for two hours.

If they were allowed to stand over night heat to almost boiling and while hot add slowly by pouring down the stirring rod, with a watch glass partially over the beaker, concentrated H_2SO_4 to a complete precipitation. Usually 25 ml. of the H_2SO_4 is sufficient to cause complete precipitation. Care must be taken in adding the H_2SO_4 in order to avoid any loss by spattering.

The samples are next cooled in a cold water bath and filtered through Whatman #1 filter paper. Wash precipitate five or six times with a 5 % H_2SO_4 solution.

Boil down clear filtrate to a volume where the salts begin to drop out solution, cool again in a cold water bath and filter off precipitated salts by using a No. 1 filter paper, washing precipitate with the 5 % H_2SO_4 wash solution.

Procedure:

Add 10 ml of 3 % H_2O_2 solution to filtrate stir well and add slowly dry Na_2CO_3 until foaming stops and the solution shows blue to litmus paper then add 2-3 gms in excess.

Cover beakers with glasses and boil for 3 to 4 minutes, remove from heat and allow to stand until precipitate of iron hydroxide has collected and settled. Filter through #1 12.5 on filter paper and wash ppt and beaker with hot distilled water which has had added 2-3 gms of Na_2CO_3 per liter. A small amount of paper pulp may be necessary in order to obtain a clear solution. A convenient size vessel to receive filtrate in is a 400 ml. beaker.

Remove filtrate from filters; discarding precipitate; and add conc. H_2SO_4 slowly until foaming ceases and add 3-5 ml in excess. Add glass beads, cover beakers with watch glasses and boil down to a volume of approx. 100 ml. or to the place where ppt. of salts begins and remove from heat. Bumping maybe cut down conveniently by the placing of a strip of filter paper on the tip of a stirring rod in the solution. Dilute sample to about 200 ml. and place back on heat. While the solutions are boiling add 50 % NH_4OH solution. The NH_4OH should be added cautiously to avoid spattering or bumping until a yellow focculant precepitate is formed and then add 5 ml. of NH_4OH in excess, giving a strong odor of ammonia. Bring back to boiling on hot plate. On removing from heat add approx. 5 ml. of cold conc. NH_4OH to solution and set aside for a few minutes until precipitate has collected and settled. When they have settled sufficiently filter through # 42 filter paper on which a little paper pulp has been added. Wash ppt. and beaker well with hot distilled water to which has been added NH_4OH to a distinct ammonia odor.

After thoroughly washing beaker and precipitate with the ammonia water add 100 ml. of a 1:10 solution of H_2SO_4 to the original beaker, warm and pour over the filter, dissolving the yellow precipitate; catching the filtrate in a 250 ml. beaker, washing the beaker and filter paper well with hot distilled water. If the samples have shown a high percentage of copper and nickel it is a good precaution to reprecipitate the yellow "T" hydrate again following the same procedure as given above.

Carbide and Carbon Chemicals
Corporation, Operating Contractor for
the U.S. Atomic Energy Commission.

PLANT RECORDS

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After the solution of the yellow precipitate of "T" has been dissolved in the 1:10 H_2SO_4 solution cool to room temperature and add dropwise a solution of 5% $KMnO_4$ until the sample shows a permanent pink color. At this stage the solutions are ready to be passed through the Jones Zinc Reductor which has previously had a blank determined on it as follows:

Take 100 ml. of the 1:10 H_2SO_4 solution which has been turned a light pink with $KMnO_4$, and run through the Reductor at the rate of some 350-400 c.c. in ten minutes. After thoroughly washing the Reductor with distilled water titrate the reduced solution with the standard $KMnO_4$ solution previously standardized. The blank should not amount to more than 0.50 ml. of the standard $KMnO_4$. If the blank should be over 0.50 ml. or not constant, the Reductor should be continually washed with 1:10 solution of H_2SO_4 and distilled water until the blank is lowered and becomes constant. (The Reductor should never be allowed to go dry before or during a determination.)

When the blank has been arrived at the unknown samples are run through the reductor into 500 ml. Erlenmeyer flask at the rate of speed as the blank and washed until the total volume is approx. 400 ml. Remove flask from the reductor and bubble air through them for 5-10 minutes thus oxidizing any of the "T" which was reduced by the reductor to below a valence of four back to that valency.

After reduction and aeration of the samples they are titrated to a faint pink coloration with the standardized solution of $KMnO_4$. One ml. of 1N solution of $KMnO_4$ is equivalent to - 0.119 gms of "T".

Calculations:

$$\frac{(\text{ml } K\text{ } mno_4 \times \text{Normality of } K\text{ } mno_4) \times 0.119}{25} = \quad \times 100 = \% \text{ "T"}.$$

1 c.c. of normal $KMnO_4$ is equivalent to 0.119 gms. "T".

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